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Soybean: For Textile Applications and Its Printing

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Abstract

It is vital to colorize sustainable, renewable, ecologic natural-based soybean fiber properly via printing for the textile and fashion industry. Optimum steaming-fixation conditions in respect of colorimetric values and color fastness properties should be determined for dye class in order to obtain the best possible print quality on soybean fiber fabric. This study exhibits that acid and 1:2 metal-complex dyes (originally used for printing of natural protein fibers such as wool and silk) and special reactive dyes (used for wool and polyamide fibers printing) can be used for regenerated soybean fiber printing leading to high color strength with adequate color fastness performance. Steaming at 102°C for 40 and 45 minutes are the optimum fixation conditions for acid and 1:2 metal-complex dyes on soybean fiber fabrics, respectively. On the other hand, steamings at 102°C for 20 minutes and 30 minutes are the optimum fixation conditions for wool-type reactive dyes and polyamide-type reactive dyes on soybean fiber fabrics, respectively. These optimum steam-fixation durations for each dye class led to the highest light fastness levels. Optimum steam fixation durations for 1:2 metal-complex and reactive dye classes (for both wool and polyamide) on printed soybean fibers displayed quite high and commercially acceptable wash fastness and good and commercially acceptable dry rub fastness and moderate to good wet rub fastness levels performance.

Keywords: soybean, soybean fiber, printing, dye, reactive dye, metal complex dye, acid dye, fixation, fastness, color

1. Introduction

Rising world population needs more amount of textile fibers from year to year leading to necessity for higher world fiber production to meet increasing world fiber demand [1]. This surplus fiber demand has been recently met by the increase of manmade fiber production from petrochemicals which are processed using highly toxic chemical methods and will not decompose naturally [2]. Moreover, increasing oil prices, descending petroleum reserves and

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rising concerns regarding ecology set off alarm bells. Therefore, researchers and textile manufacturers are seeking biodegradable, sustainable and renewable textile fiber alternatives as an effective tool for compensating the world fiber demand while reducing the influence of the textile industry on the environment due to rising consumer awareness and demands about eco-friendly and organic products [2, 3]. Soybean plant is the source for one of those promising renewable, sustainable and biodegradable fibers for more sustainable world textile production. Soybean plant is a species of legume native to East Asia and its bean is not only edible but also has many uses [4]. One of those uses is in the textile industry. The soybean plant can be used for both cellulosic- and protein-based textile fiber production [5]. The first attempts to produce textile fibers from soybean protein were carried out during the mid-twentieth century [1, 6–8]. However, there were noteworthy challenges on its production in economic quantities and on fiber performance such as fiber strength that led to a decreasing interest for soybean protein fiber at that time [5, 9]. Nonetheless, as aforementioned, at the end of the twentieth century, there was a growing attention on eco-friendly natural-based sustainable biodegradable fibers due to ecological concerns, which leads to the awakening of promising soybean protein fiber. Key technological developments also provide opportunity for soybean protein fiber production with an ecologically friendly route leading to renewed interest [10–13]. Soybean cultivation has recently become much more cost-effective and soybean is one of the most abundant agricultural crops [14]. Therefore, soybean is cheap and abundant [6]. Furthermore, recent technical performance enhancement of soybean protein fiber via geneticengineering techniques extends the commercial scope of this fiber [10, 11]. Therefore, in the 2000s, new soybean protein fiber, made from soybean protein and polyvinyl alcohol, was developed and a new soybean fibers' production process commercially promoted, standardized and launched to the textile markets [1]. The previous tenacity-related problems were also overcome with the inclusion of polyvinyl alcohol. Modern techniques for soybean fiber production make use of cutting-edge bioengineering principles by means of usable protein that is extracted from waste materials: the leftover dregs from soybean oil, tofu and soymilk production [15]. On the other hand, in the case of cellulosic-based textile fiber from soybean plant, natural cellulose fibers were produced from soybean straw by a simple alkaline extraction in 2009 and the researchers reported that these fibers exhibit similar properties and structure to natural cellulose fibers from conventional sources and natural cellulose fibers derived from soybean straws could be suitable for textile, composite and other industrial uses [2, 16].

Soybean fiber is the only protein-based botanic fiber and derived from renewable plant sources and a man-made fiber and manufactured in China in vast amount [2, 17]. Soybean fiber is manufactured from soybean protein that can be manufactured in massive quantities and at a low cost [17]. Soybean fiber is a kind of regenerative plant fiber that is created from regenerated soya *GlycineMax* soybean proteins along with polyvinyl alcohol (PVA) as a predominant component [1]. So in other way of saying, soybean fiber (SPF) is natural plant-based man-made regenerated protein fiber that is produced from a blend of soybean protein and polyvinyl alcohol [9, 18]. The soybean plant, its seeds and soybean protein fiber are shown in **Figure 1**.

Soybeans contain great quantity of proteins, approximately 37–42%, compared to peanut (25%), milk (3.2%) and corn (10%) proteins [1, 21]. Soybean proteins can be used as food, feed,

Figure 1. Soybean plant, soybean seeds and soybean fiber (SPF) [9, 19, 20].

textile fiber, pharmaceutical, ink, adhesive, emulsion, cleansing material and plastic [1, 6, 7]. Soybean proteins are globular proteins and they are composed of varied individual proteins and a large variety of molecular-sized protein aggregates [9, 18]. The most important proteins of soybean are globulins and soybean proteins have two storage proteins: glycinin (30% of the total soybean seed protein) and β-conglycinin (predominant and 30–50% of the total soybean seed) [1, 9, 18]. Soybean proteins comprise 18 amino acids and the predominant amino acid of soybean protein is glutamic acid with 18.2% share [1, 18]. In more detail, soybean proteins consist of glycine (8.8%), alanine (7.5%), phenylalanine (4.4%), valine (6.3%), leucine (9.8%), isoleucine (4.8%), serine (6.4%), threonine (4.3%), tyrosine, aspartic acid (12.8%), glutamic acid (18.2%), histidine (5.5%), arginine (0.8%), lysine (3.9%), tryptophan and proline (5.6%) [1, 18]. Moreover, soybean protein also includes little amount of sulfur containing amino acids such as cysteine (1%) and methionine (0.35%) [1]. Globular proteins comprise polypeptide segments that are linked by hydrogen and disulfide bonds and electrostatic and hydrophobic interactions [1, 18].

Liquefied soybean protein is extruded from soybean after the extraction of soybean oil and mechanically processed to manufacture soybean protein fiber by utilizing new bioengineering technology [2, 22]. The manufacturers of soybean protein fiber declared that the soybean fiber production is ecofriendly and does not impart any damage to atmosphere, environment, human body and water [9, 18]. Soybean fiber production steps are displayed in **Figure 2**. But initially, oil is extracted from soybean and residual cake from the extraction is kept aside [15]. Soybean protein is not suitable for fiber spinning owing to its globular structure and for this reason denaturation and degradation processes, which are important processes for fiber formation, are applied to soybean protein to convert the protein solution into a spinnable fiber spinning dope [18]. The denaturation process of soybean protein could be carried out with alkalis, heat, or enzymes using bioengineering techniques [18, 23, 24]. There are only conformational changes occurring in denaturation stage and in this step, the protein molecule unfolds to result in linear protein chains retaining its primary structure [18]. Subsequently protein spinning solution is prepared with polyvinyl alcohol (PVA) and protein

Figure 2. Production steps of soybean fiber [9, 13, 18, 20].

that is extracted from this residual protein cake [15] (**Figure 2**). Then, fiber spinning solution is spun using the wet spinning method. In this part, the fiber spinning dope solution, which comprises soybean and polyvinyl alcohol, is filtered and then forced through the spinneret. In the spinnerets, molecular chains are oriented and arranged into a structure involving crystalline and amorphous regions [18]. This orientation is greatly maintained in two sequential coagulation baths and after the coagulation steps, the cross-linking process is applied to soybean fibers in order to improve their mechanical properties [18] (**Figure 2**). Coagulated fibers are passed into a cross-linking bath after winding and it is reported that cross-linking step with glutaraldehyde could improve the mechanical properties of soybean protein fiber [18]. The last stages of the soybean protein fiber manufacture are washing, drying, followed by the drawing process in order to enhance the tensile strength properties of soybean fibers. Then the fiber can undergo winding, heat setting and cutting processes. Finally, soybean fibers with various specifications and varied lengths can be produced [15].

Soybean protein fiber is under the classification of Azlon group and it is also known as "vegetable cashmere," "artificial cashmere," and "soy silk" due to its cashmere feel [5, 9, 25]. The natural color of soybean protein fibers is pale yellow or cream [5, 15]. Soybean fiber merges environmental advantages with satisfactory textile performance. As aforementioned, soybean fiber is eco-friendly, sustainable and biodegradable fiber [23]. Actually, this fiber can exhibit not only numerous aesthetic qualities in association with natural fibers, but also physical features which are more akin to those of the synthetic fibers [5]. Soybean fiber is soft, smooth, light and has natural luster like silk fiber, which contributes a luxurious appearance to its fabric [17, 25]. Soybean fiber exhibits perfect draping ability leading to elegant appearance and feeling with comfortable wearing conditions [17]. Moreover, soybean fiber displays excellent moisture absorption performances like those of cotton fiber but superior ventilation and moisture transmission properties leading to perfect moisture management ability [12, 17, 25, 26]. Soybean fiber fabrics are warm and comfortable with high heat of wetting [17].

Soybean fibers possess good mechanical properties such as single soybean fiber tenacity of 3.0 cN/dtex that is higher than that of silk, wool and cotton fibers [17, 25]. Nonetheless, the wet strength of soybean fiber is 35–50% of its dry strength [17]. What is more, this fiber also displays splendid easy wash, fast-dry and crease-resistance performance [25, 27, 28]. Soybean protein fiber exhibits antibacterial resistance for *Styphalococcus aureuses*, *Coli bacillus* and *Candica albicans* [1, 25]. They also have beneficial effect on human skin and human health due to their amino acid content [15]. The amino acids of soybean protein fiber could activate the collagen protein in the human skin, resist tickling and evaporate the skin [25]. Moreover, ultraviolet radiation absorption performance is better than that of cotton, viscose and silk fibers and can reach up to 99.7% [1, 22, 25].

After all, soybean protein fiber can satisfy the performance, comfort and functional demands of conventional and technical textile goods [22]. Therefore, soybean protein fiber has many various end-use application areas in the textile industry such as nonwovens, infant clothes, apparel, t-shirt, skirt, bed linen, undergarments, sleepwear, sportswear, bed sheets, towels, blankets, etc. [18, 25]. In addition, soybean fiber can be used alone and/or in blends with cashmere, wool, cotton, silk, elastic and synthetic fibers.

There are quite few studies about the coloration, limited to dyeing process, of soybean fibers in the literature, which are dyeing with 1:2 metal-complex, acid, direct, reactive dyes and natural dyes [22, 29–34]. Choi et al. [29] investigated the performance of three acid dyes and some reactive dyes containing different reactive groups on soybean protein fiber in terms of exhaustion, fixation and build-up. In this study, monochlorotriazine (MCT), monofluorotriazine (MFT), difluorochloropyrimidine (DFCP) and vinyl sulfone (VS) reactive groups-based dyes were studied. Soybean protein fiber exhibits good dyeing brilliance and good color fastness to light and perspiration [15].

Moreover, Chongling and Zan-min [35] studied the dyeability of soybean fibers with reactive disperse dyes in the supercritical carbon dioxide environment. However, coloration is not only limited to dyeing process for textile surfaces, textile printing is also an important coloration process of applying color to the textile substrate in certain patterns and/or designs in the textile industry in order to decorate the fabric. Textile printing enables creating patterns, which could be impossible to compose with any other techniques, such as weaving and/or dyeing. It is also right spot to mention that printing is not only an important way of coloration but also a way of self-expressing styles and an important fashion tool. Therefore, it is also important to colorize this sustainable, renewable ecologic natural-based soybean protein fiber via the printing process using available commercial dyes.

In this study, coloration via printing of soybean fiber with commercial chemical dyes [acid dyes, metal-complex dyes and reactive dyes (for polyamide and wool fibers)] and the effect of different steaming durations on colorimetric and color fastness (wash, rub, light fastness, etc.) properties of printed soybean fiber were examined and discussed. The optimum conditions for printing soybean protein fiber have not been studied within the literature reviewed. Therefore, the most appropriate dye class for soybean printing and the optimum fixation durations for soybean fiber printed with each dye type were examined and determined.

Printed soybean fabric samples were fixed with different steaming times (such as 10, 15, 20, 25, 30, 40, 45 minutes) at 102°C. Color fastness (wash, rub and light fastness) and colorimetric (*K/S,* CIE *L*^{*}, *a*^{*}, *b*^{*}, *C*^{*} and *h*^{*o*} co-ordinates, reflectance spectra and CIE Chromaticity Diagram) properties were investigated and compared.

2. Materials and methods

In this study, 100% soybean fiber single-jersey knitted fabric (fabric weight of 110 $\rm g/m^2$ and yarn count of 30/1) was utilized for coloration via printing. In order to determine the most suitable dye type for soybean printing, commercial acid dyes, 1:2 metal-complex dyes and reactive dyes (for polyamide and for wool fibers) were applied to soybean fiber via the printing process. It is known that acid, metal complex, reactive and chrome dyes can be used for protein fiber (wool and silk) printing processes. However, 1:1 metal complex dyes and chrome dyes have recently lost their significance in textile printing. Therefore, acid dyes, 1:2 metal complex dyes and reactive dyes have generally been preferred for textile printing purposes. Indeed, printing of wool and silk fibers is carried out using acid, 1:2 metal complex and reactive dyes in the textile industry. Silk fibers can be printed under similar conditions to wool fibers using acid and 1:2 metal-complex dyes. In the case of the printing process with reactive dyes, wool-fiber printing can be carried out under acidic conditions; however, silk fibers can be printed under alkaline conditions due to their higher stability under alkaline conditions in comparison with wool fibers. In this study, soybean fibers were printed with different dye classes, which are recommended for silk, wool and polyamide fibers. Both blue and red dyes were used for each dye class and all dyes were supplied from Huntsman (Huntsman Corporation, USA). Dyestuff information and fixation periods (steaming at 102°C) are shown in **Table 1**. Printing processes on soybean fabrics were carried out using printing paste recipes shown in **Table 2**.

Table 1. Dye types and fixation (steaming) time after printing.

^a y: Water solubility of neutral dyeing acid dyes and metal complex dyes for printing is generally low, therefore necessary amount of hot water is carefully added to the printing pastes in order to ease the solubility of these dyes ᵇ 1:2 (ammonium sulfate solution in water; 1 part of ammonium sulfate and 2 parts of water)

Table 2. Print paste recipes of each dye classes for soybean fiber.

Viscosity degree of printing paste (**Table 2**) was measured with a No. 5 spindle using a Brookfield DV-I Prime Viscometer (20 Rpm) (DV-I PRIME, Brookfield Engineering Laboratories, USA) and measuring viscosity degree 40 poise as a base. Soybean fiber fabrics were printed at 8 m/ minutes at press 6 on using Atac laboratory-type printing machine (RGK 40, Atac, Turkey) with 70 Nr PES gauze and a doctor blade 8 mm in diameter under the laboratory conditions.

Printed soybean fiber fabrics were dried in a laboratory-type Atac drying machine (FT-200, Atac, Turkey) at 100°C for 3 minutes. Then, printed soybean fiber fabrics were steamed at 102°C for various fixation steaming durations (**Table 1**) with a laboratory-type steamer (ATC-HB350G, Atac, Turkey) for the dye fixation. It was earlier reported that optimum yields would only be acquired under humid steaming conditions when steaming prints on wool-proteinfiber fabric [36]. Moreover, it is also stated that the most brilliant and color-fast prints can only be acquired in saturated steam fixation at $100-102^{\circ}C$ [37]. Therefore, fixation of soybean regenerated protein fiber fabrics following the printing process was carried out with steaming method at 102°C. Various steaming times (**Table 1**) were applied to printed soybean fiber fabrics in order to investigate optimum fixation conditions for soybean fiber printed with each dye class. After the fixation, printed soybean fiber fabrics were washed and dried at room temperature. After printing, the effects of different printing dye types and different steaming fixation times on colorimetric and color fastness properties of printed soybean fiber fabrics were evaluated and compared.

2.1. Colorimetric measurements

The CIE *L*, a*, b*, C** and *h*^{*o*} color coordinates were measured and the *K/S* (color strength) values calculated from the reflectance values at the appropriate wavelength of maximum absorbance (λ_{\max}) for each fabrics using a DataColor SpectraFlash 600 (DataColor SpectraFlash 600, Datacolor International, USA) spectrophotometer (D65 day light, 10° standard observer). CIE color space is a color assessment technique, which compares the sample to be tested to a standard (white). Numerical data were acquired and recorded using a reflectance spectrophotometer (DataColor SpectraFlash 600, Datacolor International, USA) to obtain CIE *L*a*b** values as follows:

$$
L^*
$$
(Lightness/Darkness); Black = 0 and White = 100 (1)

On the *a** axis (red to green), positive values specify amounts of red while negative values specify amounts of green [*a**; Red = Positive Value (+*a**) and Green = Negative Value (−*a**)]. In the case of *b** axis (yellow to blue), positive numbers demonstrating increased yellowness and negative numbers demonstrating blueness [*b**; Yellow = Positive Value (+*b**) and Blue = Negative Value (−*b**)].

The *K*/ *S* values of the fibers were determined through Kubelka-Munk equation as given below:

$$
K/S = (1 - R)^2 / 2R
$$
 (2)

where *R* is the reflectance at complete opacity, *K* is the absorption coefficient and *S* is the scattering coefficient. Moreover, reflectance spectra, CIE chromaticity diagrams (CIE chromaticity diagram exhibits the mapping of human perception with regard to *x* and *y* values. Here, color is stated in regard to these two CIE parameter color coordinates; *x* and *y*.), *K/S*-*C**, *a*-b** and *L**-*C** colorimetric graphs of printed soybean fabrics were measured and presented. *h°* (hue angle) is expressed in degrees. The starting point of the hue angle is at the +*a** axis (redness) where the hue angle is 0°. The hue angle is 90° for the +*b** axis (yellowness), 180° for the −*a** axis (greenness) and 270° for the −*b** axis (blueness). Saturation (*C**: Chroma) and *h°* can be calculated according to below equations:

$$
\bigcup \bigcup \bigcup \bigcup \bigcup \bigcup \bigcup \bigcup \bigcup \bigcap \{a^*\} = \big[(a^*)^2 + (b^*)^2\big]^{1/2} \bigcup \bigcup \bigcup \bigcup \bigcup \bigeq (3)
$$

$$
h^{\circ} = \arctan(b * / a^*)
$$
 (4)

2.2. Color fastness determination

Wash, rub (dry and wet) and light fastness properties were determined according to ISO 105:C06 A2S (40°C in a M228 Rotawash machine, SDL ATLAS, UK), ISO 105: X12 and ISO 105: B02 (color fastness to artificial light: Xenon arc lamp) standards, respectively. ISO grey scale was used for the estimation of color fastness of the printed soybean fiber fabrics to washing and to dry and wet rubbing. Color fastness to light was determined using the blue-wool scale.

3. Results and discussion

Data obtained from the assessments of printed soybean fabric colorimetric properties appear in **Tables 3**–**6** and **Figures 3**–**26**, while the results of the color fastness properties of printed soybean fabrics appear in **Tables 7** and **8**.

3.1. Colorimetric properties of soybean fiber fabric printed with acid dyes (Erionyl dyes)

Soybean fiber fabrics were printed with the acid dyes that are generally used for wool and silk printing. Colorimetric data of soybean fiber fabrics after printing with acid dyes and following fixation via steaming are shown in **Table 3** and **Figures 3**–**8**. It can be easily seen that the reflectance spectra of soybean fabrics printed with Erionyl Blue A4G and Erionyl Red A3G dyes (acid dyes) and then fixed via steaming at various steaming periods were very close to

Table 3. Color coordinates of soybean fabrics printed with acid dyes (Erionyl dyes).

Table 4. Color coordinates of soybean fabrics printed with 1:2 metal complex dyes (Lanacron).

Table 5. Color coordinates of soybean fabrics printed with reactive dyes for wool (Lanasol dyes).

Table 6. Color coordinates of soybean fabrics printed with reactive dyes for polyamide (Eriofast dyes).

each other and even overlapped for some cases (**Figure 3**). Therefore, soybean fabrics printed with studied acid dyes and then fixed with steaming at different periods exhibited close colorimetric values without drastic changes (**Table 3** and **Figures 5**, **7**). Moreover, the shade differences of the visual appearances of fabrics printed with red (Erionyl Red A3G) and blue (Erionyl Blue A4G) acid dyes were also detected on reflectance spectra, a^* , b^* and h° values [**Figures 3**, **5** (CIE chromaticity diagram), 7 (*a*-b** plot) and **Table 3**]. Especially, CIE chromaticity diagram shows the exact shades of printed soybean fabric samples with their measured chromaticity coordinates on two-dimensional (*x-y*) color diagram (**Figure 5**).

Figure 3. Reflectance (%)-wavelength (nm) spectra of soybean fabrics printed with acid dyes (Erionyl dyes).

There was no big difference between the color strength values (*K/S*) of soybean fabrics printed and steamed for 15 and 30 minutes (**Table 3** and **Figure 4**). Increasing steaming time to 40 minutes on soybean fabrics led to a slight increase on color strength. However, it seems that longer steaming period such as 45 minutes was not necessary since such prolonged steaming application resulted in a slight decrease in color strength (**Table 3** and **Figure 4**). The highest color strength values for both Erionyl Blue A 4G (*K*/*S* with 17.33) and Erionyl Red A 3G (*K*/*S* with 24.26) dyes were attained after 40 minutes of steaming for fixation. It is in parallel with the literature which states that relatively long steaming times of 30–60 minutes are generally required to fix acid dyes on other protein fibers; wool or silk [37].

As it can be observed from **Figure 6**, chroma values (*C**) of soybean fabrics printed with Erionyl Blue A-4G and fixed with varying times exhibited close values. As earlier mentioned,

Figure 4. Color strength degrees of soybean fabrics printed with Erionyl acid dyes according to various fixation steaming duration.

Figure 5. CIE chromaticity diagram showing the chromaticity coordinates of soybean samples printed with the Erionyl acid dyes.

Figure 6. *K/S*-*C** (color strength versus chroma) diagram of soybean fabrics printed with Erionyl acid dyes and fixed with different steaming periods.

Figure 7. *a**-*b** (redness-greenness versus yellowness-blueness) diagram of soybean samples printed with Erionyl acid dyes.

40-minute steaming resulted in the highest color strength (*K/S* of 17.33) for fabrics printed with Erionyl Blue A-4G. On the other hand, in the case of Erionyl Red A-3G, 40 minutes of steaming led to the highest color strength (24.26) and the highest chroma (63.33) on printed soybean fabrics (**Figure 6**). It is clear that both color strength and chroma values of soybean protein fiber fabrics printed with Erionyl Red A-3G were significantly higher than those of soybean printed with Erionyl Blue A-4G (**Figure 6**).

*a** and *b** values of soybean samples printed with Erionyl Blue A-4G and fixed with varied steaming periods were very close to each other (**Figure 7**). On the other hand, in the case of Erionyl Red A-3G, 40-minute steaming resulted in redder and yellower appearance with a

Figure 8. *L**-*C** (Lightness versus chroma) diagram of soybean fabrics printed with Erionyl acid dyes and fixed with different steaming periods.

slightly higher *a** value and a slightly higher *b** value in comparison to other steamed samples (**Table 3** and **Figure 7**). Lightness (*L**) and chroma (*C**) degrees of soybean fabrics printed with Erionyl Blue A-4G and fixed with varying times exhibited close values (**Figure 8**). The 40-minute steamed sample exhibited the lowest lightness value of 34.82 leading to the highest color strength of 17.33, as expected (**Figure 8** and **Table 3**). The higher color strength (*K/S*) led to the lower lightness values (*L**). In the case of Erionyl Red A-3G dye, as earlier mentioned, the 40-minute steamed sample displayed the highest chroma value (**Figure 8**).

3.2. Colorimetric properties of soybean fiber fabric printed with 1:2 metal complex dyes (Lanacron dyes)

Colorimetric data of soybean fiber fabrics after printing with acid dyes followed by fixation via steaming are shown in **Table 4** and **Figure 9**–**14**.

It is clear that the reflectance spectra of soybean fabrics printed with Lanacron Blue 3GL and Lanacron Red 2GL dyes (1:2 metal complex dyes) and then fixed via steaming at various steaming periods were very close to each other (**Figure 9**). Prolonged steaming time on soybean fabrics printed with 1:2 metal complex dyes (Lanacron Blue 3GL and Lanacron Red 2GL dyes) resulted in an increase in color strength for both dyes (**Table 4** and **Figure 10**). The highest color strength values were observed for 45-minute steamed soybean samples printed with both Lanacron Blue 3GL (*K*/*S* of 24.65) and Lanacron Red 2GL (*K*/*S* of 23.05) dyes (**Table 4**). It is known that the steam, used after printing, provides the moisture and rapid heating, which gives rise to the transfer of dye molecules from the thickener film (guar-based thickener in our case) to the fiber within a reasonable time [37]. It seems that prolonged steaming time resulted in better fixation and higher attachment rates of the 1:2 metal complex dyes on the soybean fiber leading to higher color strength in general.

Figure 9. Reflectance (%)-wavelength (nm) spectra of soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes).

Figure 10. Color strength degrees of soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes) according to various fixation steaming durations.

The color shade differences of the visual appearances of soybean protein fiber fabrics printed with Lanacron Blue 3GL and Lanacron Red 2GL dyes (1:2 metal complex dyes) were also detected on reflectance spectra, CIE chromaticity diagram, *a*-b** plot and hue angle *(h*^o) values (**Figures 9**, **11**, **12** and **Table 4**). Particularly CIE chromaticity diagram displayed the exact shades (red and blue colors) of printed soybean fabric samples with their measured chromaticity coordinates on two-dimensional (*x*-*y*) color diagram (**Figure 11**). Soybean samples printed with 1:2 metal complex dyes and fixed with varied steaming periods (15, 25, 30, 40, 45 minutes) exhibited very close *a** and *b** values (**Figure 12**).

Lightness (*L**) and chroma (*C**) degrees of soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes) and fixed with varying times exhibited close values (**Figure 13**). Printing with metal complex dyes generally results in duller color prints with less brightness [37]. For instance, soybean fabrics printed with Lanacron Red 2GL were brighter than samples printed

Figure 11. CIE chromaticity diagram showing the chromaticity coordinates of soybean samples printed with 1:2 metal complex dyes (Lanacron dyes).

Figure 12. *a**-*b** (redness-greenness versus yellowness-blueness) diagram of soybean samples printed with 1:2 metal complex dyes (Lanacron dyes).

with Lanacron Blue 3GL (**Figure 13**). It is known that color brightness increases while *C** and *L** values are both rising at the same time [38]. Acid dyes (Erionyl dyes) led to brighter appearance on soybean fabric in comparison with 1:2 metal complex dyes (Lanacron dyes). Indeed, higher lightness (*L**) and higher chroma (*C**) values were measured in the case of acid dyes (Erionyl Blue A 4G and Erionyl Red A 3G) when compared to 1:2 metal complex dyes

Figure 13. *L**-*C** (Lightness versus chroma) diagram of soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes) and fixed with different steaming periods.

Figure 14. *K/S*-*C** (color strength versus chroma) diagram of soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes) and fixed with different steaming periods.

(Lanacron Blue 3GL and Lanacron Red 2GL) (Tables 3, 4 and **Figures 8** and **13**). Chroma values (*C**) of soybean fabrics printed with Lanacron Blue 3GL and Lanacron Red 2GL and fixed with varying times displayed close values (**Figure 14**). As aforementioned, color yield (*K*/*S*) of printed soybean samples increased with increased fixation periods. It seems that the proper diffusion of large 1:2 metal complex dye molecules into the soybean fiber needs time and the diffusion increases with the increased steaming fixation times leading to a high color yield.

Figure 15. Reflectance (%)-wavelength (nm) spectra of soybean fabrics printed with reactive dyes for wool (Lanasol dyes).

3.3. Colorimetric properties of soybean fiber fabric printed with reactive dyes for wool (Lanasol dyes)

Colorimetric data of soybean fiber fabrics after printing with reactive dyes (for wool) followed by fixation via steaming are shown in **Table 5** and **Figure 15**–**20**. It is clearly observable that the reflectance spectra of soybean fabrics printed with Lanasol Blue 3R reactive dye and then fixed via steaming at various steaming periods were very close to each other and even overlapped for some cases (**Figure 15**). Soybean fabrics printed with Lanasol Blue 3R and then fixed with steaming at different periods exhibited close colorimetric values without drastic changes (**Table 5** and **Figure 16**, **18**–**20**). On the other hand, the reflectance spectra of soybean fabrics printed with Lanasol Red 5B reactive dye and then fixed via steaming at various steaming periods were slightly different leading to slightly different color properties (**Table 5** and **Figure 16**, **18**–**20**).

There was no big difference between the color strength values (*K/S*) of soybean fabrics printed with Lanasol Blue 3R dye and then fixed via steaming at various steaming periods (**Figure 16** and **Table 5**). There were differences between the color strength values (*K/S*) of printed with Lanasol Red 5B dye and steamed soybean fabrics (**Figure 16** and **Table 5**). The highest color strength values for both Lanasol Red 5B (*K/S* with 24.78) and Lanasol Blue 3R (*K/S* with 9.89) dyes were obtained after 20 minutes steaming for fixation. Longer steaming periods such as 25 or 30 minutes slightly decreased color strength (**Figure 16** and **Table 5**).

The color shade differences of the visual appearances of soybean protein fiber fabrics printed with Lanasol Blue 3R and Lanasol Red 5B dyes (reactive dyes for wool) were also detected on CIE chromaticity diagram, *a**-*b** plot and hue angle *(h*^o) values (**Figure 17**, **18** and **Table 5**). It is known that reactive dyes constitute true chemical bonds with the SH, NH, or NH₂ groups in the polypeptide chains in acid media (pH 3–5) at 80–100°C and these dyes can provide brilliant color in prints [37]. Particularly CIE chromaticity diagram displayed the exact shades (maroon and dark blue colors) of printed soybean fabric samples with their measured chromaticity coordinates on two-dimensional (*x*-*y*) color diagram (**Figure 11**). Soybean samples

Figure 16. Color strength degrees of soybean fabrics printed with reactive dyes for wool (Lanasol dyes) according to various fixation steaming durations.

Figure 17. CIE chromaticity diagram showing the chromaticity coordinates of soybean samples printed with reactive dyes for wool (Lanasol dyes).

printed with Lanasol Blue 3R and fixed with varied steaming periods (10, 15, 20, 25, 30 minutes) exhibited very close *a** and *b** values (**Figure 18**). In the case of Lanasol Red 5B dye, 10-, 15-, 25- and 30-minute steamed soybean samples were slightly redder and bluer in comparison with 20-minute steamed soybean sample according to *a** and *b** values (**Figure 18**).

Lightness (*L**) and chroma (*C**) degrees of soybean fabrics printed with Lanasol Blue 3R and fixed with varying times exhibited close values (**Figure 19**). In the case of Lanasol Red 5B dye, 10-, 15-, 25- and 30- minute steamed soybean samples, when compared to 20-minute steamed soybean sample, exhibited slightly higher chroma and higher lightness leading to slightly brighter appearance (**Figure 19**). As aforementioned chroma values (*C**) and color yields (*K/S*)

Figure 18. *a**-*b** (redness-greenness versus yellowness-blueness) diagram of soybean samples printed with reactive dyes for wool (Lanasol dyes).

Figure 19. *L**-*C** (lightness versus chroma) diagram of soybean fabrics printed with reactive dyes for wool (Lanasol dyes) and fixed with different steaming periods.

of soybean fabrics printed with Lanacron Blue 3GL and fixed with varying times displayed very close values (**Figure 14**). Overall, the highest color strength values (*K/S*) for both Lanasol reactive dyes were obtained after 20 minutes of steaming. It is known that reactive dyes for printing wool possess better solubility than acid dyes and can be usually sprinkled directly into the print paste as solids without the use of dye solvents and that they need shorter steaming times which are a clear benefit in continuous steaming [39]. This is clearly in line with the results of soybean fabrics printed with reactive dyes for wool (Lanasol dyes), since, in this case, short steaming time as 20 minutes was enough for satisfying print quality from the color point of view. However, one should be careful while working with reactive dyes in printing, since unlevelness problem in large blotches can occur in some shade areas [39].

Figure 20. *K/S*-*C** (color strength versus chroma) diagram of soybean fabrics printed with reactive dyes for wool (Lanasol dyes) and fixed with different steaming periods.

Figure 21. Reflectance (%)-wavelength (nm) spectra of soybean fabrics printed with reactive dyes for polyamide (Eriofast dyes).

3.4. Colorimetric properties of soybean fiber fabric printed with reactive dyes (Eriofast dyes)

Soybean fiber fabrics were printed with the reactive dyes (Eriofast dyes), which are generally recommended for polyamide printing. Colorimetric data of soybean fiber fabrics after printing with reactive dyes (Eriofast dyes) followed by fixation via steaming are shown in **Table 6** and **Figures 21**–**26**. It can be easily seen that the reflectance spectra of soybean fabrics printed with Eriofast Red B reactive dyes and then fixed via steaming at various steaming periods were slightly different leading to slightly different color properties (**Table 6** and **Figures 21**, **23**–**26**).

On the other hand, Eriofast Blue 3R printed and fixed with various steaming periods soybean samples exhibited closer reflectance spectra leading to close color properties (**Table 6** and **Figures 21**, **23**–**26**). Prolonged steaming time in soybean fabrics printed with Eriofast dyes (reactive dyes for polyamide) led to an increase in color strength for both dyes (**Table 6** and **Figure 22**). A similar case was also observed for 1:2 metal complex dyes. The highest color strength values were observed for 30-minute steamed soybean samples printed with both

Figure 22. Color strength degrees of soybean fabrics printed with reactive dyes for polyamide (Eriofast dyes) according to various fixation steaming durations.

Figure 23. CIE chromaticity diagram showing the chromaticity coordinates of soybean samples printed with reactive dyes for polyamide (Eriofast dyes).

Eriofast Blue 3R (*K*/*S* of 14.35) and Eriofast Red B (*K*/*S* of 17.20) dyes (**Table 6**). It could be said that prolonged steaming time caused better fixation and higher attachment rates of Eriofast reactive dyes in the soybean fiber leading to higher color strength.

The color shade differences of the visual appearances of soybean protein fiber fabrics printed with Eriofast Blue 3R and Eriofast Red B dyes (reactive dyes for polyamide) were also detected on reflectance spectra, CIE chromaticity diagram, a^* -b^{*} plot and hue angle (h^o) values (**Figures 21**, **23**, **24** and **Table 6**). Particularly CIE chromaticity diagram displayed the exact shades (red and blue colors) of printed soybean fabric samples with their measured chroma-

Figure 24. *a**-*b** (redness-greenness versus yellowness-blueness) diagram of soybean samples printed with reactive dyes for polyamide (Eriofast dyes).

Figure 25. *L**-*C** (lightness versus chroma) diagram of soybean fabrics printed with reactive dyes for polyamide (Eriofast dyes) and fixed with different steaming periods.

ticity coordinates (**Figure 23**). Soybean samples printed with Eriofast Blue 3R reactive dye and fixed with varied steaming periods (10, 15, 20, 25 and 30 minutes) displayed close *a** and *b** values (**Figure 24**). A similar observation could be made for Eriofast Red B reactive dye with one exception. Only 20-minute steamed soybean fabric printed with Eriofast Red B dye was slightly redder and yellower due to higher *a** and *b** values (**Figure 24** and **Table 6**).

Figure 26. *K/S*-*C** (color strength versus chroma) diagram of soybean fabrics printed with reactive dyes for polyamide (Eriofast dyes) and fixed with different steaming periods.

Soybean fabrics which are printed with Eriofast reactive dyes and fixed with varying times exhibited close lightness (*L**) and chroma (*C**) values with slight differences (**Figure 25**). 20-minute and 30-minute steamed samples exhibited the highest chroma values for Eriofast Red B (59.3) and Eriofast Blue 3R (40.4), respectively (**Figure 25**). 30-minute steamed samples exhibited the lowest lightness values leading to the highest color strength, as expected (**Figure 25** and **Table 6**). Soybean fabrics printed with Eriofast Red B were brighter than soybean fabrics printed with Eriofast Blue 3R with higher lightness and chroma levels (**Figure 25**). Soybean fabric printed with Eriofast Red B and fixed with 30-minute steaming displayed the highest color strength and chroma value (**Figure 26**). For Eriofast Red B, 30 minutes of steaming resulted in the highest color strength value.

3.5. Color fastness properties of printed soybean fabrics

Color fastness of colored material is a very important factor for buyers' demand [40, 41]. Color fastness is the resistance of color to fade or bleed of colored textile substrates occurring due to various types of influences such as water, light, rubbing, washing, perspiration, etc., which normally occur in textile manufacturing and in our daily use [41, 42]. Washing and light fastness properties are the most important parameters to evaluate the performance of the textile material and to decide its end-use application type [43]. In addition, dry and wet rub fastness properties are also an important for apparel applications [17]. The effects of dye-class type and fixation time by steaming on the color fastness properties of soybean fiber fabrics printed with commercial dyes are discussed below. Wash, rub (dry and wet) and light fastness properties of printed soybean samples are shown in **Tables 7** and **8**.

3.5.1. Light fastness

It seems that increase in steaming time resulted in very slight light fastness performance improvement in some cases (**Table 7**). This observation is quite visible in the case of Eriofast dyes (reactive dyes for polyamide). In this case, prolonged steaming fixation times resulted in up to three quarter point improvement on light fastness values. This is most probably due to their higher color strength leading to higher dye content in the fiber. Although acid dyes resulted in vibrant colors on soybean fibers, their related light fastness values were not so high and in the range of 4–4/5 and 3–3/4 for Erionyl Blue A-4G and Erionyl Red A-3G dyes, respectively (**Table 7**). A 1:2 metal complex dyes (Lanacron dyes) led to the highest light fastness performance of seven rating with only very slight fading on soybean fabrics according to the blue-wool scale (**Table 7**). These quite high light fastness levels are not surprising, since metal complex dyes are known to impart higher fastness properties in comparison with acid dyes [37]. However, on the other hand, metal complex dyes may result in duller colors [37]. Indeed, both measured brightness and light fastness differences between soybean fabrics printed with acid and 1:2 metal complex dyes were in line with this previous experience. Reactive dyes which are recommended for wool fibers (Lanasol dyes) resulted in moderate to good light fastness values on soybean fibers with 4/5–*5*/6 which are higher than the light fastness levels of acid dyes (Eriofast dyes). Other studied reactive dyes which are recommended for polyamide fibers (Eriofast dyes) caused slightly higher light fastness levels on soybean fibers with 5–5/*6* in comparison with reactive dyes for wool fibers (Lanasol dyes) (**Table 7**). These good light fastness

Printed soybean fabrics [dye class, dye name, fixation (steaming) time]		K/S	Light fastness		Rub fastness (X12)	
			$(Xenon) (1-8)$	Dry	Wet	
	Eriofast Blue 3R, 30 min	14.35	$5 - 6$	$4 - 5$	4	
	Eriofast Red B, 10 min	13.10	5	$4 - 5$	$3 - 4$	
	Eriofast Red B, 15 min	15.41	$5 - 6$	$4 - 5$	$3 - 4$	
	Eriofast Red B, 20 min	16.81	$5 - 6$	$4 - 5$	$3 - 4$	
	Eriofast Red B, 25 min	16.81	$5 - 6$	$4 - 5$	$3 - 4$	
	Eriofast Red B, 30 min	17.20	$5 - 6$	$4 - 5$	4	

Table 7. Light and rub fastness properties of printed soybean fabrics.

results were not surprising since reactive dyes generate true chemical bonds with the SH, NH, or NH₂ groups in the polypeptide chains of the protein fiber leading to good fastness levels and brilliant colors [37]. Optimum steam fixation durations, which were reported and discussed in the color properties section for each dye class led to the highest light fastness levels. This is most probably owing to the higher color strength (*K/S*) with higher dye content in the fiber.

3.5.2. Rub fastness

In analogy with the light fastness performance, the lowest rub fastness levels were obtained for acid dyes, as expected (**Table 7**). The dry and wet rub fastness levels of soybean printed with Erionyl Blue A-4G acid dyes were in the range of 3–4 and 2–3, respectively. Erionyl Red A-3G dyes resulted in up to 1 point improvement for both dry (*4*/5–4/5) and wet (3–4) rub fastness when compared to Erionyl Blue A-4G (**Table 7**). It is known that wool and silk protein fibers printed with acid dyes exhibit very vivid print colors with moderate fastness levels. Therefore, acid dyes must be selected to achieve acceptable light and wet fastness for each end-use, along with the preferred brilliance of hue [37].

Soybean fabrics printed with 1:2 metal complex dyes (Lanacron dyes) exhibited 3–4 gray scale rating for wet rub fastness. In the case of dry rub fastness, blue dye (Lanacron Blue 3GL) resulted in commercially acceptable fastness levels of 4–5 gray scale rating which was about half point higher than those of red dye (Lanacron Red 2GL) (**Table 7**). Reactive dyes, which are recommended for wool fibers (Lanasol dyes), led to moderate to good rub fastness levels on soybean fibers with 3/4–4 for dry rub and 4–*5* for wet rub fastness (**Table 7**). Other studied reactive dyes, which are recommended for polyamide fibers (Eriofast dyes) resulted in similar rub fastness levels on soybean fibers with 3/*4*–4 for dry rub and 4–*5* for wet rub fastness (**Table 7**). It is expected that reactive dyes for printing wool protein fiber exhibit good wet fastness properties [39]. A 1:2 metal complex dyes and reactive dyes (for both wool and polyamide) resulted in quite good and commercially acceptable dry rub fastness and moderate to good wet rub fastness levels. The different steaming times did not result in significant differences on rub fastness level. Prolonged steaming fixation times sometimes resulted in only up to a quarter point improvement on wet rub fastness value.

3.5.3. Wash fastness

Printed soybean samples for all dye classes and all steaming times exhibited commercially acceptable wash fastness levels, which are equal to or above 4 gray scale rating (**Table 8**). Most of them were gray scale rating of 5 with no staining at all. The rests exhibited only one point lower wash fastness levels than the maximum available (**Table 8**). Although acid dyes resulted in slightly lower wash fastness levels than other three dye classes, wash fastness levels of soybean fabrics printed with acid dyes are still good and in the commercially acceptable range. A 1:2 metal complex dyes and reactive dyes (for both wool and polyamide) led to quite good and commercially acceptable wash fastness levels. As mentioned earlier, reactive dyes can form covalent bonds with $-NH$, $-NH_{2}$, $-SH$ and $-OH$ groups of protein fibers leading to high fastness levels. There were no significant differences between the wash fastness levels due to different dye class, different dye and different fixation steaming time. The different steaming times did not result in significant differences on wash fastness level. Prolonged steaming fixation times sometimes resulted in only up to a quarter point difference on wash fastness value.

4. Conclusions

It is important to colorize sustainable, renewable ecologic natural-based soybean fiber properly via printing for the textile and fashion industry. Dye selection and fixation conditions after printing affect the color yield and quality of the print. Optimum fixation conditions in respect of colorimetric values and color fastness properties should be determined for dye class in order to obtain the best possible print quality on soybean fiber fabric. In the case of soybean protein fabrics printed with acid dyes (Erionyl dyes), the highest color strength values for Erionyl Blue A 4G (*K/S* = 17.33) and Erionyl Red A 3G (*K/S* = 24.26) dyes were obtained after 40 minutes of steaming for fixation. In the case of 1:2 metal complex dyes (Lanacron dyes), the highest color strength values were observed for 45-minute steamed soybean samples with both Lanacron Blue 3GL (*K/S* = 24.65) and Lanacron Red 2GL (*K/S* = 23.05) dyes. These two observations are in parallel with the literature where it was stated that relatively long steaming times of 30–60 minutes are generally required to fix acid and metal complex dyes on wool and silk protein fibers. It is known that the steam used after printing provides the moisture and rapid heating which gives rise to the transfer of dye molecules from the thickener film to the fiber within a reasonable time. It seems that the proper diffusion of the large 1:2 metal complex dye molecules into the soybean fiber needs a little more time and the diffusion increases with increasing steaming fixation time leading to a high color yield. Acid dyes (Erionyl dyes) led to brighter appearance on soybean fabric in comparison with 1:2 metal complex dyes (Lanacron dyes). The highest color strength values for Lanasol Red 5B (*K/S* = 24.78) and Lanasol Blue 3R (*K/S* = 9.89) dyes (Bromo acrylamide reactive group reactive dyes which are generally recommended for wool printing) on soybean were obtained after 20-minute steaming fixation. In the case of novel sulfo group containing Eriofast reactive dyes which are generally recommended for polyamide printing, the highest color strength values were observed for 30-minute steamed soybean samples with Eriofast Blue 3R (*K/S* = 14.35) and Eriofast Red B (*K/S* = 17.20) dyes. It is known for printing wool protein fiber that reactive dyes possess better solubility than acid dyes and can be usually sprinkled directly into the print paste as solids without the use of dye solvents and that they need shorter steaming times which is a clear benefit in continuous steaming.

Light fastness values of soybean printed with acid dyes were not so high and in the range of 4–4/5 and 3–3/4 for Erionyl Blue A-4G and Erionyl Red A-3G dyes, respectively. A 1:2 metal complex dyes (Lanacron dyes) led to the highest light fastness performance of 7 rating with only very slight fading on soybean fabrics. Reactive dyes which are recommended for wool and polyamide fibers (Lanasol and Eriofast dyes) resulted in moderate to good light fastness values on soybean fibers with 4/5–*5*/6 and 5–5/*6*, respectively, which were higher than the light fastness levels of acid dyes (Eriofast dyes). In analogy with the light fastness performance, the lowest rub fastness levels were obtained for acid dyes. A 1:2 metal complex dyes and reactive dyes (for both wool and polyamide) on soybean printing resulted in quite good and commercially acceptable dry rub fastness and moderate to good wet rub fastness levels. The different steaming times did not result in significant differences on rub fastness level. Prolonged steaming fixation times sometimes resulted in only up to a quarter point improvement on wet rub fastness value. Printed soybean samples for all dye classes and all steaming times exhibited commercially acceptable wash fastness levels, which are equal to or above 4 gray scale rating. Acid dyes resulted in slightly lower wash fastness levels than other three dye classes. A 1:2 metal complex dyes and reactive dyes (for both wool and polyamide) on soybean printing led to quite good and commercially acceptable wash fastness levels. Reactive dyes can form covalent bonds with $-NH$, $-NH$ ₂, $-SH$ and $-OH$ groups in the polypeptide chains of protein fibers leading to high fastness levels. The different steaming times did not result in significant differences on wash fastness level. Prolonged steaming fixation times sometimes resulted in only up to a quarter point difference on wash fastness value.

This study exhibits that acid and 1:2 metal-complex dyes (originally used for printing of natural protein fibers such as wool and silk fibers) and special reactive dyes (used for printing of wool and polyamide fibers) can be used for the printing process of regenerated soybean fiber leading to high color strength with adequate color fastness performance. Steaming at 102°C for 40 and 45 minutes are the optimum fixation conditions for acid and 1:2 metalcomplex dyes on soybean fiber fabrics, respectively. On the other hand, steamings at 102°C for 20 minutes and 30 minutes are the optimum fixation conditions for wool-type reactive dyes and polyamide-type reactive dyes on soybean fiber fabrics, respectively. These optimum steam-fixation durations for each dye class led to the highest light fastness levels. This is most probably owing to their higher color strengths (*K/S*) with higher dye content in the fiber. Overall, optimum steam fixation durations for 1:2 metal complex and reactive dye classes (for both wool and polyamide) on printed soybean fibers displayed quite high and commercially acceptable wash fastness and good and commercially acceptable dry rub fastness and moderate to good wet rub fastness levels performance.

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